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 (72) Inventor JOHN WILTON



(54) HEAT-INSULATING SHAPED COMPOSITIONS

(71) We, FOSECO TRADING A.G., a Swiss Company of Langenjohnstrasse 9, Postfach, Chur 2, Graubunden, Switzerland, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to heat-insulating shaped compositions and their production and particularly to such compositions which contain a high melting refractory ingredient. It further relates to apparatus for use in the production of such shaped products.

In recent years there have been developed heat-insulating shaped compositions for use as slabs or sleeves for lining hot tops or the heads of ingot moulds in the foundry and the related industries. Such compositions may contain, for example, fibrous cellulosic material, granular refractory material and a binding agent and optionally may contain a proportion of fibrous refractory material. Slabs, sleeves or the like of such compositions are commonly made by forming a slurry of the ingredients and introducing this into a vessel having one or more walls formed of a mesh, and pressing the slurry onto the mesh so that the liquid passes through as effluent and the solids are retained in a compacted body of the required shape adjacent to the mesh. The slabs, sleeves and the like so obtained then have to be dried out and are usually stoved to increase their strength.

Whilst the foregoing method has been found superior to all others in its general principles in that by means of its slabs and sleeves of excellent quality may be obtained, the method is not without its drawbacks. In particular the time taken to de-water the slurry is rather long especially when the fibrous content is relatively high and the slabs or sleeves formed contain a great deal of the slurry liquid so that a con-

siderable drying operation is required.

Compositions commercially made by the said process usually have a density of about 1 g/cc or more and a porosity of 7-12 AFS units. It is desirable to have lower densities than this since products of lower density, higher porosity, have improved heat-insulating properties. However attempts to achieve this have commonly met with difficulty in that the product has tended to have mechanical strength and refractoriness below the acceptable levels.

Slabs and sleeves have also been used for many years, for the above end uses, i.e. for hot-tops or risers or the head of ingot moulds, made usually of a granular refractory, e.g. sand, and a bonding agent such as a resin. These have also suffered from the difficulty that, in general, if steps are taken to try to increase porosity, i.e. to reduce their density, the products have tended to lack mechanical strength or refractoriness.

According to the present invention there are provided slabs sleeves and the like which comprise refractory calcium silicate fibre, coke in particulate form, and a binder therefor, the proportion of calcium silicate fibre being 11-70% by weight.

It is preferred that the calcium silicate fibre should have a melting point at least 1100°C and preferably at least 1400°C, i.e. a continuous operating temperature of at least 750°C, preferably 800°C. Fibrous calcium silicates (e.g. slag wools, rock wools) have melting points in the range 900°C-1350°C and are suitable for use at a continuous operating temperature of up to about 800°C. However, in applications where resistance to high temperature is only required for a few hours, operating temperatures of up to 1400°C are permissible. The maximum operating temperature does however, depend to some extent upon the purity of the calcium silicate fibre since the

presence of any alkali metal compounds decreases the melting point of the fibre appreciably. Naturally, the calcium silicate fibre may be employed in the manufacture of lining materials for casting moulds in which metal is cast at temperatures higher than the continuous working temperature since they are required to withstand such temperatures for only a short period because the molten metal commences cooling immediately upon casting.

A typical example of calcium silicate fibre which can be used is the following.

mineral wool:

| | | |
|-----------|--------------------------------|----------|
| analysis: | CaO | 30-40% |
| | SiO ₂ | 30-40% |
| | Al ₂ O ₃ | 10-15% |
| | MgO | 6-8% |
| | MnO | about 1% |

as well as traces of other oxides. Maximum continuous working temperature 760-810°C.

In some applications, notably for use in casting copper and cast iron, it is desirable to increase the refractoriness of the slabs and sleeves. This may be done, for example, by treating the calcium silicate fibre with a chromate solution, followed by heat treatment, by which means chromic oxide is introduced as part of the chemical composition of the fibre; such processes are described in Canadian Patent Nos. 564,670 and 564,671. Other chemical treatments involve ion-exchange procedures whereby elements such as calcium, sodium and potassium may be replaced by aluminium or chromium. Improved refractoriness may also be obtained by incorporating in the composition a proportion of milled or chopped spun organic fibres e.g. rayon, cotton, jute, acrylonitrile. These appear to carbonise when heated in the absence of air and this increases the refractoriness of the composition.

Another means of increasing the overall refractoriness of the shaped products involves applying to the surfaces of the shape a refractory coating e.g. an aqueous suspension of zirconia flour.

The binder may be any material known for use for bonding refractory materials. The particular binder selected generally depends on the process by which the sleeve is made, and this is discussed further below.

Sleeves, slabs and the like according to the present invention are preferably made by felting the calcium silicate fibre onto a mesh former from a slurry. By this means slabs and sleeves may be produced easily and quickly and of low density, for example of density 0.35 gm/cc or even less.

According therefore to a further feature of the present invention there is provided a process for the production of a slab, sleeve or the like of the type described above which comprises forming a slurry comprising calcium silicate fibre, particulate cake

and a binder therefor, the calcium silicate fibre constituting 11-70% by weight of the solids content of the slurry and wherein said solids comprise 0.2%-5.0% by weight of said slurry, wholly immersing in said slurry a former having at least one wall formed of mesh material and having a lead-away pipe, said former being stationary in said slurry, establishing a subatmospheric pressure within the pipe which is between 1.5 and 5 psi below atmospheric pressure, to suck away the liquid of the slurry through the mesh and away through the lead-away pipe, while depositing on the mesh surface a compacted layer of the suspended materials, removing the former from the slurry while maintaining the subatmospheric pressure in the lead-away pipe, removing the compacted layer from the former and stoving the resulting slab, sleeve or the like to drive off the remaining slurry liquid.

The slurry is preferably aqueous.

It is possible to operate using a slurry which contains no binder to produce coherent slabs and sleeves but in the interest of obtaining a high mechanical strength in the final product some binder is included. Since if soluble binders are used a great amount may be lost in the effluent from the process of the invention they are for that reason less suitable. Moreover it has been found that a soluble binder is capable of migration within the formed slabs so that, if a said slab is stoved, as is conventional, the product may have a hard skin but a rather weak interior. For these reasons solid, substantially insoluble binders, dispersed in the slurry, are generally preferred. Suitable such materials are urea- and phenol-formaldehyde resins, clays such as bentonite and china clay, starch, wheat flour, phosphate-modified starches and colloidal silica. Mixtures of soluble and insoluble binders may be used to provide good mechanical strength and surface hardness.

The solids content of the slurry is 0.2% to 5% by weight. At higher contents it becomes more difficult to control the process to yield products of the desired thickness and density.

By a suitable choice of materials and other variable conditions of the process of this invention, it is possible to operate at relatively low suction pressures, i.e. 1.5 to 5 p.s.i. By reason of the fact that the slurry solids deposit to form a highly permeable slab, large quantities of liquid may be sucked very rapidly through the mesh. Thus for example it has been found possible to produce sleeves of $\frac{1}{2}$ " thickness from a 2% by weight aqueous slurry in 5-10 seconds, using only $1\frac{1}{2}$ p.s.i. suction pressure. Using this slurry technique, the particulate cokes is preferably predominantly of particle size in the range -100 +300 mesh BSS sieve. If

there is too great a proportion of very fine particles these tend to be lost by passing through the perforated former with the effluent. Furthermore, if the particle size distribution is very wide, a higher packing density will result, producing products of higher specific gravity and requiring longer times of greater suction pressures to effect dewatering. A suitable sieve grading for the coke is one with no more than about 25% minus 300 mesh, the remainder being in the above-quoted range of $-100 + 300$ mesh.

The slabs, sleeves and the like made by the process of the invention may be stoved to increase their mechanical strength if desired. The products obtained may have a very low density, e.g. 0.2 gms per cc or less, whilst the porosity may be as high as 300 AFS units.

The invention further includes apparatus for use in carrying out the process of the invention which comprises a tank adapted to hold a slurry (the slurry tank), a perforate former connected by a conduit to a vacuum pump and adapted to be removable from, and wholly immersed in, the slurry in the slurry tank, and when so immersed to be static in the tank, means for discharging water from the conduit into a reservoir tank, means for discharging water from said reservoir tank into a tank filled with a stirring device and adapted for the formation of a slurry therein (the slurry formation tank) and means for the discharge of slurry from said slurry formation tank into said slurry tank, the pump being so constructed and arranged to effect withdrawal of water from the slurry tank through the perforate former when the latter is below the level of the slurry in the slurry tank and withdrawal of air through the conduit when the perforate former is removed from the slurry tank.

A specific embodiment of such apparatus is illustrated in the accompanying drawing. Referring to this drawing, the apparatus comprises a slurry tank 1, a perforated former 2 shown immersed in the tank, and conduit 3 leading from the former 2 via a reservoir and water trap 6 to a pump 5. A stop cock 4 is provided in the exit line from reservoir and water trap 6 to a further reservoir tank 7. The apparatus also includes a slurry formation tank 11 provided with a stirrer 8. A pump 9 is provided between tanks 7 and 8, and a further pump 10 between tank 8 and tank 1, and the various elements of the apparatus are interconnected with piping as shown in the drawing. If desired a single pump may be provided with appropriate valves and cocks to perform the functions of the several pumps shown in the drawing.

In use, a slurry is charged into the tank 1 and the former 2 is immersed in it. The pump 5 is brought into operation causing

liquid to be sucked from the slurry tank 1 via the former 2, through the conduit 3 so that the solids of the slurry are deposited on the former. The reservoir 6 may discharge the withdrawn liquid into tank 7 by opening stop cock 4, whence it is discharged as required into tank 11. Further solid ingredients for the slurry are introduced into tank 11 and slurried in the liquid so introduced. The resultant slurry is used as a feed to the slurry tank 1. Thus the liquid is recycled and the solid constituents added to tank 11 are such as to constitute that which is deposited on the former 2.

When the desired thickness of solids is deposited on the former by the action of the apparatus as just described the former is raised clear of the slurry in the slurry tank. Continuing operation of the pump 5 causes withdrawal of liquid from the deposit carried by the former. This is important since without the continuation of the suction pressure as thus described the deposit on the former may tend to disintegrate as soon as it is stripped from the former as a shaped product.

The shaped deposit may be stripped from the former by any convenient method, e.g. it may be subjected to slight pressure to deform and loosen it from the former, it may be shaken free by vibration or it may be ejected by reversing the air flow of pump 5.

The formed shapes are of course still wet and should be dried, e.g. by placing them in an oven.

The following Examples in which all parts and percentages are by weight will serve to illustrate the invention:—

EXAMPLE 1

The fibrous refractory material used in this example was a calcium silicate mineral wool of melting point Ca 815°C and analysing thus: CaO , 38%; Al_2O_3 , 14%; SiO_2 , 36%; MgO , 8% with four per cent miscellaneous impurities (TiO_2 , MnO , Fe_2O_3 and the like).

A slurry of 1% by weight solids content was made, the solid material dispensed in the slurry having the following composition by weight:—

| | |
|------------------------|-------------|
| Calcium silicate fibre | |
| (as above) | 57% |
| Crushed coke | 35% |
| Resin binder | 7% |
| | (see below) |

The resin binder used in this Example was a 1:2 mixture of urea-formaldehyde and phenol formaldehyde resins.

A former consisting of a cylindrical chamber having mesh walls on its curved surface was connected by a hose to a vacuum pump, the apparatus being as illustrated in the accompanying drawing.

The former was immersed in the slurry

and a suction pressure of 1.5 p.s.i. applied via the pump. This was continued for 8 to 10 seconds, water being sucked away at the rate of 35 gallons per minute. There was thus formed on the former a sleeve of height 6 inches, outside diameter 5 inches and inside diameter 4 inches.

The sleeves thus made were stripped from the former by reversing the air flow and then dried, a drying time of only 1-2 hours being found to be sufficient. The product has a density of 0.27 to 0.33 gm/cc.

A sleeve prepared as just described was used to line the feeder head in casting cast iron, which was poured at 1350°C and the cooling curve of the casting was determined. It was found that the sleeve gave a 20 minutes delay between the pouring of the metal and its solidification, this being more than double that which was found when using conventional sleeve of bonded sand.

If, in the foregoing example, a higher suction pressure is employed, e.g. 3 to 5 p.s.i. there is a tendency for the products to be more compact, of higher density and, therefore, possibly of inferior heat-insulating properties.

EXAMPLE 2

Various sleeves were made up by the process described in Example 1 having compositions in the ranges of:

| | |
|---|--------|
| coke dust (-100 mesh BSS) ... | 30-55% |
| calcium silicate fibre | 40-60% |
| urea-formaldehyde/phenol-formaldehyde resin mixture (1:2) ... | 4-8% |

These sleeves were found to be of particular value in casting copper and copper base alloys.

EXAMPLE 3

Sleeves having the following composition, produced by the method described in Example 1, performed well as riser sleeves for cast iron castings.

| | |
|---|-----|
| calcium silicate fibre | 37% |
| coke dust (-100 mesh BSS) | 55% |
| urea-formaldehyde/phenol-formaldehyde mixture (1:2) | 8% |

WHAT WE CLAIM IS:—

1. Slabs, sleeves and the like formed of a composition which comprises calcium silicate fibre, particulate coke and a binder therefor, the proportion of calcium silicate fibre in the composition being 11-70% by weight.

2. Slabs, sleeves and the like according to claim 1 wherein the binder is selected from urea and phenol formaldehyde resins, bentonite, china clay, starch, wheat flour, phosphate-modified starch and colloidal silica.

3. Slabs, sleeves and the like according to claim 1 or 2 wherein the calcium silicate fibre is, prior to its incorporation into the composition, treated with a chromate solu-

tion followed by a heat treatment.

4. Slabs, sleeves and the like according to any of claims 1-3 wherein the composition contains a proportion of milled or chopped organic fibre.

5. Slabs, sleeves and the like according to claim 4 wherein the organic fibre is rayon, cotton, jute or acrylonitrile.

6. Slabs, sleeves or the like according to any of claims 1-5 which are at least in part coated with a refractory dressing.

7. Slabs, sleeves and the like according to claim 6 wherein the refractory dressing is a suspension of zircon flour.

8. Slabs, sleeves and the like substantially as hereinbefore described with reference to any one of the foregoing specific Examples:

9. A process for the production of a slab, sleeve, or the like according to claim 1 which comprises forming a slurry comprising calcium silicate fibre, particulate coke and a binder therefor, the calcium silicate fibre constituting 11 to 70% by weight of the solids content of the slurry, and said solids comprising 0.2% - 5% by weight of said slurry, wholly immersing in said slurry a former having at least one wall formed of mesh material and having a lead-away pipe said former being stationary in said slurry, establishing a subatmospheric pressure within the pipe, which is between 1.5 and 5 p.s.i. below atmospheric pressure, to suck away the liquid of the slurry through the mesh and away through the lead-away pipe, while depositing on the mesh surface a compacted layer of the suspended materials, removing the former from the slurry while maintaining the subatmospheric pressure in the lead-away pipe, removing the compacted layer from the former and stoving the resulting slab, sleeve or the like to drive off the remaining slurry liquid.

10. A process according to claim 9 wherein the slurry is aqueous.

11. A process according to claim 9 substantially as hereinbefore described with reference to the foregoing specific Example 1.

12. Apparatus for use in carrying out the process of claim 9 which comprises a tank adapted to hold the slurry (the slurry tank), a perforate former connected by a conduit to a vacuum pump and adapted to be removable from, and wholly, immersed in, the slurry in the slurry tank, and when so immersed to be static in the slurry tank means for discharging liquid from the conduit into a reservoir tank, means for discharging liquid from said reservoir tank into a tank fitted with a stirring device and adapted for the formation of a slurry therein (the slurry formation tank) and means for the discharge of slurry from said slurry formation tank into said slurry tank, the

5 pump being so constructed and arranged to effect withdrawal of liquid from the slurry tank through the perforate former when the latter is below the level of the slurry in the slurry tank and withdrawal of air through the conduit when the perforate former is removed from the slurry tank.

13. Apparatus according to claim 12

substantially as hereinbefore described with reference to the accompanying drawing. 10

V. GALLAFENT & CO.,
Agents for the Applicants,
Chartered Patent Agents,
8 Staple Inn,
London. W.C.1.

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